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भाग 33 आयोडाईड

(दूसरा पुनरीक्षण)

Indian Standard

METHODS OF SAMPLING AND TEST (PHYSICAL AND CHEMICAL) FOR WATER AND WASTEWATER

PART 33 IODIDE

(Second Revision)

ICS 13.060.50

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

May 2009 **Price Group 2**

Environment Protection and Waste Management Sectional Committee, CHD 32

FOREWORD

This Indian Standard (Part 33) (Second Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Environment Protection and Waste Management Sectional Committee had been approved by the Chemical Division Council.

Iodine is naturally present in water. The average concentration in seawater is about 60 ppb, but varies from place to place. Rivers usually contain about 5 ppb of iodine, and in mineral sources some ppm can even be found. Brown algae accumulate up to 0.45 percent (dry mass) of iodine. Corals, sea sponges, shells and fishes accumulate high iodine concentrations, usually in the shape of thyroxin or tri-iodine thyroxin.

Iodine is often applied to purify water. Its primary application is drinking water disinfection. Iodine may be removed from water by means of active carbon.

The first revision of this standard was published in 1988 by superseding **25** of IS 3025 : 1964 'Methods of sampling and test (physical and chemical) for water used in industry'. In this revision, volumetric method has been incorporated to satisfy the demand of the industries.

The composition of the Committee responsible for formulating this standard is given in Annex A.

In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'.

Indian Standard

METHODS OF SAMPLING AND TEST (PHYSICAL AND CHEMICAL) FOR WATER AND WASTEWATER

PART 33 IODIDE

(Second Revision)

1 SCOPE

1.1 This standard (Part 33) prescribes two methods for determination of iodide, namely (a) Spectrophotometric method, and (b) Volumetric method in water and wastewaters.

1.2 Volumetric method shall be followed as a referee method in case of difference of opinion.

2 REFERENCES

The standards listed below contain provisions, which through reference in this text, constitute provisions of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below:

IS No.	*Title*
3025 (Part 1) : 1987	Methods of sampling and test (physical and chemical) for water and waste water: Part 1 Sampling (*first revision*)
7022 (Part 1) : 1973	Glossary of terms relating to water, sewage and industrial effluents, Part 1
7022 (Part 2) : 1979	Glossary of terms relating to water, sewage and industrial effluents, Part 2

3 TERMINOLOGY

For the purpose of this standard, definitions given in IS 7022 (Part 1) and IS 7022 (Part 2) shall apply.

4 SAMPLING AND PRESERVATION

Sampling and sample preservation shall be done as prescribed in IS 3025 (Part 1).

5 SPECTROPHOTOMETRIC METHOD

5.1 Principle

Iodide can be determined in water by using its ability to catalyze the reduction of cerium (IV) by arsenious acid. For given reaction conditions and *p*H, the catalytic effect is proportional to concentration of iodide. At the end of the fixed reaction time, the reaction is stopped by cooling. The concentration of ceric ion remaining is determined by measuring the absorbance of the solution at 520 nm and comparing with a calibrated graph based on the absorbance of iodide standard solution treated similarly. The detection limit is 5 µg/l.

5.2 Interference

The formulation of non-catalytic forms of iodine and inhibitory effects of silver and mercury are reduced by adding excess of sodium chloride that sensitized the reaction. The interference of organic materials that can be oxidized by cerium (IV) is removed by digesting sample in an alkaline medium.

5.3 Apparatus

5.3.1 *Spectrophotometer* — For use at wavelength of 520 nm and provided with a light path of 1 cm.

5.3.2 *Stop Watch*

5.3.3 *Test Tubes* — 2 × 15 cm.

5.4 Reagents

5.4.0 *Purity of the Reagents*

Unless specified otherwise, only pure chemicals and tannin free distilled water shall be used in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the result of analysis.

5.4.1 *Sodium Chloride Solution*

Dissolve 200.0 g of sodium chloride in water and dilute to 1 l. Recrystallize the sodium chloride if an interfering amount of iodine is present, using water ethanol mixture.

5.4.2 *Arsenious Acid* — 0.1 N.

Dissolve 4.946 g of arsenic trioxide in water containing 0.20 ml of concentrated sulphuric acid. Dilute to 100 ml.

5.4.3 *Potassium Hydroxide* — 2 N.

5.4.4 *Zinc Sulphate* — 100 g/l.

5.4.5 *Ceric Ammonium Sulphate* — 0.02 N.

Dissolve 12.64 g of ceric ammonium sulphate [$Ce(NH_4)_4(SO_4)_4.2H_2O$] in water, add 44 ml of concentrated sulphuric acid and make up to 1 litre.

5.4.6 *Potassium Chlorate Solution Saturated*

5.4.7 *Stock Iodide Solution*

Dissolve 261.6 mg of anhydrous potassium iodide in water and dilute to 1 000 ml; 1.00 ml = 200 µg of iodide.

5.4.8 *Intermediate Iodide Solution*

Dilute 50 ml of stock iodide solution to 1 000 ml with water; 1.00 ml =10.00 µg of iodide.

5.4.9 *Standard Iodide Solution*

Dilute 10 ml of intermediate solution to 1 litre; 1 ml = 0.1µg of iodide.

5.5 Procedure

5.5.1 *Sample Pre-treatment*

To eliminate interference from organic materials capable of being oxidized the samples are digested in an alkaline medium. To a 6 ml sample placed in a test tube, add 1 ml of zinc sulphate, 2 ml of 2 N potassium hydroxide and 2 drops of a saturated solution of potassium chlorate solution, and mix. Evaporate to 105°C until dry. Heat for 30 min at 500°C, then for 1 h at 600°C. After cooling, add 6 ml of distilled water and centrifuge for 15 min. If the solution is not turbid, it can be used for analysis. It may be necessary to clarify further by filtration.

5.5.2 A 2 ml volume of sample, standard or blank having a potassium hydroxide concentration of 0.66 N is placed in a test tube. The iodide content should be 0.005 to 0.100 µg. The test tube is placed in ice water bath and the sample permitted to equilibrate after which 2.0 ml of arsenious acid solution, ceric ammonium sulphate solution are added and solution is carefully mixed. The sample is then placed in a water bath at 40°C for exactly 20 min. The reaction is then stopped by placing the test tube in ice water bath where it is allowed to stand for exactly 10 min. After this period, measure the absorbance at 520 nm using distilled water as a reference. The absorbance of the blank is subtracted from that of the sample to obtain the net absorbance of the sample.

5.6 Calibration Graph Preparation

Add 33 ml portions of 2 N potassium hydroxide solution to each of a series of 100 ml volumetric flasks. Prepare standards and blank by adding 0 to 50.0 ml portions of the iodide working solutions and diluting to 100 ml. Aliquots of these are treated in the same manner as are samples except that they need not be subjected to the digestion procedure. Plot a graph absorbance *versus* concentration.

5.7 Calculation

$$\text{Iodide (as I), mg/l} = \frac{m}{v}$$

where

m = µg of iodide equivalent from the calibration standard curve, and

v = volume in ml of the sample.

6 VOLUMETRIC METHOD

6.1 Principle

The iodides are oxidized with bromine water to iodine which is then titrated against standard thiosulphate solution.

6.2 Reagents

6.2.1 *Sodium Carbonate*

6.2.2 *Methyl Orange Indicator Solution* — Dissolve 0.1 g of methyl orange in distilled water and dilute to 1 litre.

6.2.3 *Standard Sulphuric Acid* — 2 N.

6.2.4 *Bromine Water*

6.2.5 *Sodium Sulphite Solution* — 1 percent, *w/v*.

6.2.6 *Phenol Solution* — 5 percent, *w/v*.

6.2.7 *Potassium Iodide Solution* — 10 percent, *w/v*.

6.2.8 *Standard Sodium Thiosulphate Solution* — 0.005 N.

6.2.9 *Starch Indicator Solution*

Triturate 5 g of starch and 0.01 g of mercuric iodide with 30 ml of cold distilled water and slowly pour it with constant stirring into 1 litre of boiling distilled water. Boil for 3 min. Allow to cool and decant off the supernatant clear liquid.

6.3 Procedure

Evaporate 0.5 to 2 litres of the sample to dryness after addition of small quantity of sodium carbonate. Boil the residue thus obtained with distilled water, transfer to filter and wash thoroughly with hot distilled water. Dilute the alkaline filtrate to a definite volume. Neutralize an aliquot of the alkaline filtrate to methyl orange with standard sulphuric acid. Add bromine water drop-wise from a burette in an amount equivalent to 20 mg of bromine. After a few minutes, destroy greater portion of remaining free bromine by adding sodium sulphite solution drop wise while mixing. Wash down neck and sides of the flask with distilled water and complete the removal of free bromine by addition of 1 to 2 drops of phenol solution. Add 1 ml of standard sulphuric acid and 5 ml of potassium iodide solution and titrate liberated iodine with standard sodium thiosulphate solution adding 1 ml of starch indicator near the end of the titration. Correct the determination for blank on reagents.

6.4 Calculation

$$\text{Iodide (as I), mg/l} = \frac{1\,000\, V_1\, f}{V}$$

where

V_1 = volume in ml of standard sodium thiosulphate solution used in the titration,

f = factor representing the aliquot of the alkaline filtrate taken for titration, and

V = volume in ml of the sample taken for the test.

ANNEX A

(*Foreword*)

COMMITTEE COMPOSITION

Environment Protection and Waste Management Sectional Committee, CHD 32

Organization	*Representative(s)*
In personal capacity (*40/14, C.R. Park, New Delhi 110019*)	Prof Dilip Biswas (***Chairman***)
Bhabha Atomic Research Centre, Mumbai	Dr (Shrimati) G. G. Pandit Shri I. V. Saradhi (*Alternate*)
Bharat Heavy Electricals Limited, Haridwar	Dr N. G. Shrivastava
Cement Manufacturers Association, New Delhi	Dr K. C. Narang
Central Fuel Research Institute, Dhanbad	Dr L. C. Ram
Central Leather Research Institute, Chennai	Dr S. Rajamani
Central Pollution Control Board, Delhi	Dr S. D. Makhijani Dr C. S. Sharma (*Alternate* I) Dr S. K. Tyagi (*Alternate* II)
Central Road Research Institute, New Delhi	Shrimati Anuradha Shukla
Confederation of Indian Industry, New Delhi	Ms Seema Arora Dr. S. Majumdar (*Alternate*)
Crop Care Federation of India, New Delhi	Shri P. N. Parmeshawaran Shri Chinmay Shah (*Alternate*)
Delhi College of Engineering, Delhi	Dr Rakesh Mehrotra Dr (Shrimati) Anubha Mandal (*Alternate*)
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Department of Civil Engineering, Indian Institute of Technology, New Delhi	Dr Mukesh Khare Dr Atul Mittal (*Alternate* I) Dr Arvind Nema (*Alternate* II)
Deputy General Factory Advice Service & Labour Institute, Mumbai	Shri S. S. Gautam Shri M. R. Rajput (*Alternate*)
Directorate General of Health Services, New Delhi	Dr (Shrimati) Madhuri Sharma
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Federation of Indian Chambers of Commerce and Industry, New Delhi	Shrimati Antara Roy

Organization	*Representative(s)*
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Hindustan Lever Limited, Mumbai	Shri B. B. Dave Shri Aditya Jhavar (*Alternate*)
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Ministry of Non-conventional Energy Sources, New Delhi	Shri Vinod Kumar Jain
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Municipal Corpopation of Greater Mumbai, Mumbai	Deputy City Engineer Civil (Envt) Executive Engineer (Emissn Inventory Group) (*Alternate*)
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National Institute of Occupational Health, Ahmedabad	Dr V. Krishna Murthy Dr A. K. Mukherjee (*Alternate*)
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SGS India Limited, Chennai	Shri S. Ravi
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GMGIPN—106 BIS/ND/2009—300 Copies